CHEMICAL ANALYSIS OF ENGINEERING ALLOYS

BY
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ARMOUR INSTITUTE OF TECHNOLOGY

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Entitled

"Chemical Analyses of Engineering Alloys".

by - George C. Wilsnack and Willbur W. Patrick.

For the Degree of Chemical Engineer.

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CHEMICAL ANALYSES OF ENGINEERING ALLOYS.

by

George C. Wilsnack and Willbur W. Patrick.

The principal elements used in the engineering alloys are cadmium, copper, bismuth, lead, iron, aluminum, nickel, zinc, antimony, and phosphorus. These may be found in a large variety of combinations with one another; the exact composition of any particular alloy depending upon the nature of the work for which it is intended. It is apparently impossible, in a paper of this length, to give a separate method of analysis for each mixture of two or more metals in combinations obtainable from the above eleven elements; consequently, it is assumed in the following pages that all these elements are present in a single alloy. Although such an alloy is entirely hypothetical and impractical it is obvious that if accurate results are obtained throughout the different stages of a method which comprises the determination of very nearly all of the metals in presence of each other, a submitted sample containing from two to seven of these elements, by a consequent elimination of steps, can scarcely be expected to offer greater difficulties. It is not to be understood that these methods

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are absolutely the best which can be obtained at the present day, but they are undoubtedly as regards rapidity and accuracy by far the best of the available schemes that have been brought to the attention of the writers during their efforts to obtain a reliable method for this class of analyses. With a few exceptions no originality pertaining to any method is claimed, but the composite general scheme here presented is, at least to the authors' knowledge, not in vogue at the present time.

Volumetric methods have been given the preference wherever possible, chiefly on account of the rapidity with which they can be performed when a large number of determinations are at hand, and because the error of weighing up impurities, silicon dioxide, etc., will be eliminated.

T I N

Place an amount of sample corresponding to .5 gram or less of tin into a small casserole and add .15 gram of C. P. metallic antimony if as much or more is not already present in the alloy. If the sample contains less than this amount it is neces-

sary to make up the deficiency.

Cover the sample with 8 to 10 cc of concentrated nitric acid and take to dryness. It is advisable in order to facilitate the subsequent filtration to bake strongly for about 15 minutes. Cool, add 20 cc of dilute nitric acid, and after breaking up the lumps with a stirring rod, heat to boiling, filter, and wash several times with hot water. If the baking has been conducted properly no traces of oxides will be discernable in the filtrate. Precipitate and filter paper are then placed in a 500 cc Jena Erlenmeyer flask and treated with 20 cc of concentrated sulphuric acid and 2 grams of potassium sulphate, after which the flask is placed on a piece of asbestos over a medium hot burner and heated carefully till the first violent reaction is over. Towards the end of the oxidizing operation, while the liquid is still black however from the free carbon present, the flask and contents should be cooled and the sides of the container washed down with a stream of water. The particles adhering so firmly to the wall of the flask that the water does not loosen them should be

brought back into the main solution with the aid of a "policeman". The removal of the carbon should by no means be hastened by the addition of any oxidizing agent since this will cause the antimony to assume the pentavalent form which must be avoided. The addition of the water will now have a tendency to again cause frothing when the heat is applied making it necessary to conduct this operation very carefully. From 2 to 2 1/2 hours are usually required, depending upon the intensity of heat employed, to rid the solution completely of the carbon. Cool, dilute the sulphuric acid solution with 10 cc of water, add 90 cc of concentrated hydrochloric acid and then 78 cc more of water. Prepare a rubber stopper, that fits the neck of the flask, with four holes. Through one hole pass a glass tube bent at right angles reaching to the bottom of the flask and through the hole diagonally opposite insert another tube bent in a similar manner but extending only a short distance below the bottom of the stopper. Through the third hole pass a long thin glass rod fitted with a 2" piece of platinum wire. This rod should fit rather

snugly without, however, giving any difficulty to the operator in raising or lowering it during the determination. The platinum wire is now bent into the shape of a hook and a piece of very nearly C. P. iron wire attached. Swedish iron in plates of 1/8" thickness cut into strips of 1/8" in width and four inches in length, curled up in the form of a spiral, will suit the purpose well. Through the fourth hole pass a 1/2" tube 4" long and drawn out at the lower end to an opening small enough to enable the operator to introduce iodine solution in sufficiently small quantities to obtain the starch iodide reaction within one drop of titrating solution. The tapered end is bent slightly in the direction of the submerged wire for the purpose of conduction a stream of water to free the iron from the adhering tin solution after it has been withdrawn from the liquid. This delivery tube should also be provided with a 1/4" hole about 3/4" above the lower opening to afford a means of escape for the carbon dioxide gas which is being rapidly introduced during the titration. A carbon dioxide generator is connected to the long tube while

a piece of rubber tubing connects the short tube opposite, to a 100 cc pipette which dips into a reservoir of water. This automatically prevents any air from entering the system when a partial vacuum is formed in the flask during the determination, which cannot be taken care of by the entering carbon dioxide gas. Having attached the iron to the platinum wire, the rubber stopper is firmly placed in position in the neck of the flask and the rod lowered so that the spiral of wire rests on the bottom of the flask. The generator and water seal are attached as previously described, a cap is placed over the opening of the large delivery tube and a rapid stream of carbon dioxide gas passed through the system. After a few minutes it may safely be assumed that all of the air is expelled from the flask, whereupon heat is applied and the contents permitted to boil for 35 minutes. The solution when first placed in the flask appears a deep yellow or orange depending, of course, upon the constituents present, but shortly before commencing to boil it becomes colorless and finally black due to the liberated

metallic antimony. After the reduction is complete, remove the burner, plunge the flask into a large container of cold water and allow to remain until thoroughly cold. The iron wire is then raised to a position directly below the end of the delivery tube. The cap of this tube is then removed and with all possible haste a stream of cold freshly boiled water permitted to play upon the spiral to free it from any adhering tin solution, after which the wire is raised so that the titrating solution will not come in contact with it. Freshly prepared starch is added and without interrupting the passage of gas, standard iodine solution introduced into the flask as fast as the burette will permit. When near the end point, which is indicated by the reluctant disappearance of the blue, the titration can be carried on very slowly until completed. The blue of the starch iodide is not permanent, disappearing after a minute or two, which fact does not however detract from the accuracy of the determination.

It is not to be understood that the tin is completely reduced by the direct action of the iron

alone, neither will the addition of pulverized metallic antimony bring about the desired result. However, this metal as deposited is in a state where its chemical activity is at a maximum, thus enabling it to fully complete the reduction.

Standardize the sodium thiosulphate, which serves to determine the strength of the iodine solution, very carefully against a bichromate or permanganate solution of known strength. It is essential that the utmost accuracy be employed in the above standardizarion, since 1/10 cc more or less of a N/10 oxidizing solution makes a considerable difference in the ultimate tin value of the iodine. It has been found advisable in finding the normality of a freshly prepared iodine solution to check the theoretical standardization against C. P. tin.

If very large percentages of antimony are present, enough to obscure the end point obtained with the starch solution, it is well to treat in the usual manner up to the point where the metallic antimony separates out. The solution should then be rapidly cooled, filtered with suction and washed with hot water.

About .10 gram of metallic antimony, which has previously been dissolved in a few cc of sulphuric acid, is then added, the solution returned to the original container, and the reduction continued. The concentration of the solution after this operation should not be changed to any great extent, the additional acid introduced off-setting in a large measure the water used for washing. Antimony present up to 25% will not obscure the end point provided a good light and white background are available.

ANTIMONY

Place 1/2 to 1 gram sample, depending upon the amount of antimony present, in a samll casserole and cover with 10 cc of concentrated nitric acid.

Place the dish over a medium hot source of heat and take to dryness, exercising care to prevent loss by spattering. When completely dry increase the heat and bake for 15 minutes longer. After cooling, add 10 cc 1-1 nitric acid, break up the lumps of oxide with a stirring rod and bring to a boil. Remove the casserole from the source of heat, dilute

with water and filter, examining the filtrate carefully for any particles of oxides which may have passed through the paper. After washing a few times with hot water place filter and precipitate in a 500 cc Erlenmeyer flask, add 2 grams of potassium sulphate and cover with 20 cc of concentrated sulphuric acid. Use exactly the same precautions with regard to reducing the oxides as specified in the tin method, using particular care to remove all the adhering particles from the sides of the flask into the main solution while there is yet free carbon from the filter paper present. When the solution is perfectly clear, cool and add 50 cc of water and 10 cc of concentrated hydrochloric acid. Replace over the source of heat, permit the contents to boil for a few seconds, add exactly 110 cc of water, cool, and titrate with standard potassium permanganate solution. To standardize the permanganate solution dissolve .2 gram of the C.P. metal and proceed in the manner just described. To obtain a value of approximately .005 gram antimony per cubic centimeter of titrating solution, add 3.05 grams of permanganate to a liter of water.

PHOSPHORUS

For this element from .5 to 2 grams of the sample should be used depending upon the percentage of phosphorus present.

Place the finely divided sample in a nickel or iron crucible of about 20 cc capacity, mix intimately with a quantity of sodium peroxide sufficient to fill the crucible two-thirds full and place over a Bunsen burner. The temperature should be carefully regulated so that the fusion will be completed in seven minutes but should not be intense enough to cause mechanical losses due to the violence of the chemical reaction. Cool and place the crucible and fusion in a #2 beaker, cover with water, add hydrochloric acid in slight excess, and wash out the crucible thoroughly. Introduce a small piece of metallic aluminum and heat very carefully for a short time, until the metals have coagulated out in a sponge-like mass. After placing a small piece of aluminum in the apex of the paper, filter and wash with hot water. Any black specks noticeable after the fusion has been dissolved are to be disregarded, as they have no bear-

ing upon the result. The filtrate is transferred to a 500 cc Erlenmeyer flask and 3 grams of ammonium nitrate added. It is then heated to 85 deg. C. and ammonium molybdate added to precipitate the phosphorus. From this point the usual alkali titration method is available.

GENERAL METHOD FOR BISMUTH, CADMIUM, COPPER, LEAD, NICKEL, ZINC, IRON, AND ALUMINUM

LEAD

Weigh up a one gram sample and treat with 20 cc of hydrochloric acid and a few crystals of potassium chlorate. In the presence of large percentages of lead it is sometimes very difficult to effect solution and several additions of acid and chlorate will be found necessary. Evaporate the solution until the salts begin to crystalize out so that the volume is somewhat less than 5 cc. Dissolve the salts by diluting with 225 cc of hot water. Disregard any white precipitate which may appear upon dilution and pass hydrogen sulphide to saturation. It is essential that the acidity as given be strictly adhered to as otherwise cadmium will be incompletely precipitated

and small amounts will come down as a hydrate in the nickel determination. Filter the sulphides with suction, wash well with hot water, and place the filtrate upon the hot plate to evaporate to a convenient volume for future use. The paper is placed in a #2 beaker, covered with nitric acid and 10 cc sulphuric acid and taken to copious fumes of sulphur trioxide. After the carbon has been entirely oxidized the solution is cooled and diluted to 225 cc with hot water. Again pass hydrogen sulphide, filter with suction on an asbestos pad, wash with hot water, and add this filtrate to the previous one. The precipitate which now contains tin, antimony, copper, bismuth, cadmium, and very nearly all the lead, is placed with the asbestos in a #2 beaker and covered with 30 cc of water. Add about 8 grams of potassium hydrate and allow to boil for 5 minutes. This will dissolve the tin and antimony sulphides, leaving the copper, cadmium, bismuth, and lead unaffected. Wash well with boiling water. It is essential that the washing operation be carried on in a thorough manner to rid the precipitate from the tin and antimony salts. The mechanical inclusion of these two salts should be reduced to

a minimum as otherwise the future precipitate of the two metals as oxides will be contaminated with comparatively large amounts of copper, etc. Discard the filtrate, place the asbestos and precipitate in a #2 beaker and cover with concentrated nitric acid and a little hydrochloric acid. After dissolving the sulphides, filter off the asbestos, wash well with hot water and evaporate the filtrate to a small volume. Add 5 cc of nitric acid and very carefully take to a bake. When cool add 5 cc of dilute nitric acid, bring to a boil, add 10 cc of hot water and filter off any antimony or tin oxides which may have separated out. Wash well with hot water and reject the precipitate. Add 5 cc of concentrated sulphuric acid to the filtrate and take to fumes of sulphur trioxide. Dilute with 225 cc of water and allow to stand for about 4 hours. Filter the lead sulphate on a weighed Gooch crucible and wash with a 3% sulphuric acid solution. Set the crucible aside for the time being as the precipitate does not represent the total lead present in the sample.

BISMUTH

Neutralize the filtrate from the lead with ammonia, cool, and add a mixture of 50 cc of a 3% hydrogen peroxide solution and 30 cc of ammonia. Allow the precipitated bismuth peroxide to settle and filter on a paper with suction. Wash with a small quantity of a mixture of 2 volumes of hydrogen peroxide, 1 volume ammonia, and 8 volumes of cold water, then with warm 1-8 ammonia, and finally with hot water. Place the filtrate over a source of heat to evaporate somewhat for future use. Precipitate and paper are placed in a #2 Jena beaker and covered with the usual quantity of nitric acid and 5 cc sulphuric acid and taken to fumes. Dilute with a small amount of water and filter off any silicon dioxide which may have accumulated during the determination. Wash well with hot water and dilute up to about 225 cc with cold water. Cool and precipitate the bismuth for the second time by neutralizing and treating exactly as described above. This second precipitation will assure the operator of a precipitate free from any other salts. The bismuth peroxide precipitate is filtered on a weighed

Gooch crucible and washed as described above and the filtrate united with the previous one. During the ignition of the precipitate, care should be taken to keep the reducing portion of the flame some distance from the bottom of the crucible, as otherwise a reduction to the metal will occur. The full flame should be applied for at least 25 minutes after which the precipitate and crucible are allowed to cool and the bismuth weighed as the oxide.

COPPER

Neutralize the filtrate from the bismuth which has been evaporating on the hot plate and which should now have a volume of about 300 cc, with dilute sulphuric acid. Add 3 or 4 drops of acid in excess, warm slightly, and pass hydrogen sulphide to precipitate both the cadmium and copper as sulphides. Filter with suction, wash well with hot water, and discard the filtrate. Place the precipitate in a Jena beaker, cover with nitric acid and add 5 cc sulphuric acid, take to fumes, cool, dilute up to about 30 cc with water, and, if silicon dioxide is suspected to be present, filter. Neutralize the solution with

ammonia, and add two or three drops of dilute sulphuric acid, this amount having been found sufficient to give the required acidity for the copper determination. Bring to a boil and add 4 cc of hypophosphorous acid. Reduce the volume by evaporation until the mass becomes pasty, dissolve the salt in a minimum amount of hot water, add an additional cubic centimeter of the same reagent, and again take down to a pasty consistency. Dilute to about 250 cc and filter off the metallic copper, washing well with hot water. Set the filtrate aside for the cadmium determination. The copper and filter paper are placed in a porcelain crucible and ignited. When the paper has been completely oxidized, transfer the copper oxide to a #1 beaker and remove the last traces of copper from the crucible by the addition of hot nitric acid. Carefully cover the oxide with the same acid and evaporate to about 1 cc. Cool, add 5 cc each of water and ammonia, making the solution strongly ammoniacal. Boil for exactly one minute and again cool. Add 6 cc of acetic acid and transfer the solution to a 250 cc Erlenmeyer flask.

Cool, add 2 grams of potassium iodide in solution and agitate violently for about 30 seconds. Titrate the liberated iodine with a standard solution of sodium thiosulphate. When near the end point add starch and again shake the flask violently, whereupon the titration can be completed. The sodium thiosulphate is standardized against C. P. copper in an exactly analagous manner.

CADMIUM

The filtrate from the copper is neutralized with ammonia, made one drop acid with sulphuric, diluted if necessary to a volume of 300 cc and heated to about 50 deg. C. Saturate with hydrogen sulphide and after the precipitate has settled filter on a weighed Gooch crucible by means of suction. It is first washed with hydrogen sulphide water, and then successively with hot water, carbon disulphide, alcohol, and ether. The precipitate is dried below 100 deg. C., and weighed as cadmium sulphide.

LEAD

It is a very difficult matter to control the acid sulphide separation to such an extent as to abso-

lutely prevent small amounts of lead from remaining in solution after passing hydrogen sulphide. It is, therefore necessary that this trace of lead be recovered at this point to make the separation complete. Transfer the filtrate from the acid sulphide group which has been evaporating to a #2 beaker. Evaporate to fumes, cool, dilute with water to about 150 cc and filter, washing well with a 3% sulphuric acid solution. The filtration should be carried on in the Gooch crucible containing the main portion of the lead sulphate previously obtained. Ignite carefully and calculate to lead.

NICKEL

Cool the filtrate from the lead thoroughly and add a cold concentrated solution of sodium hydroxide until a permanent precipitate has formed. Again cool, and add with constant stirring a concentrated solution of alkali containing 15 grams of sodium hydroxide. The total volume should now be from 150 to 200 cc. Place the beaker and contents over a burner, bring to a boil and add a slight excess of bromine water, after which the solution is permitted

to boil 5 minutes longer. Allow the precipitate to settle and filter, washing well with hot water. The black nickel oxy-hydroxide carries down with it from .3 to .7% of zinc, depending, of course, upon the percentage of this element present. It is, therefore, necessary to redissolve the hydrate in a little hot dilute hydrochloric acid and reprecipitate in the manner just described. Join the filtrates and permit to evaporate for the subsequent zinc determination. The precipitated nickel oxy-hydrate is again redissolved in 10 cc of hot dilute hydrochloric acid. Neutralize with ammonium carbonate in the cold and add one gram of ammonium acetate. Place on the hot plate and allow to boil vigorously for a few minutes, after which the precipitate of iron and aluminum is filtered off. This is the regular acetate separation practiced in mineral analyses. Wash the precipitate well with hot water, after which acidify the filtrate with 10 cc of concentrated hydrochloric acid, place on the hot plate and allow to boil in order to expell the acetic acid. The iron and aluminum precipitates are redissolved in a small quantity of hot dilute

hydrochloric acid. Reprecipitate with ammonia and boil to remove the excess. Now add four drops of ammonia and filter immediately. Wash well with hot water and join the filtrate with the previous one. Set the precipitate aside for the time being to be united with the remainder of the iron and aluminum hydrates which will be obtained later. To avoid confusion we will call this precipitate "A". To the filtrate now containing only the nickel, add 5 cc of dilute nitric acid and 16 cc of dilute sulphuric acid. Neutralize with ammonia and bring the volume up to about 200 cc with cold water, cool, and titrate in the usual manner with silver nitrate and potassium cyanide solutions.

ZINC

The filtrate from the hydrates of nickel, iron, and aluminum which contains all of the zinc and some of the aluminum as sodium aluminumate is made nearly neutral with concentrated hydrochloric acid and a solution of sodium carbonate added in slight excess. Allow to boil for a short while and filter off the zinc carbonate and aluminum hydrate which have pre-

cipitated. Reserve the filtrate for any aluminum which may have escaped precipitation at this point due to the presence of too great an excess of alkali. It is well to make this filtrate acid and place on the hot plate to evaporate to a volume more convenient for handling. The carbonate and the hydrate precipitates are dissolved in hot dilute hydrochloric acid and 3 grams of ammonium chloride added. The solution is neutralized with ammonia, boiled to expell the excess, removed from the source of heat, four drops of ammonia added, and the aluminum filtered off. Redissolve the aluminum hydrate in a small amount of hot dilute hydrochloric acid. Again add 3 grams of ammonium chloride and reprecipitate in the same manner. Set this aluminum hydroxide precipitate, which we will call "B", aside for the present. The filtrate is neutralized and 3 cc of concentrated hydrochloric acid added in excess. Bring to a boil and titrate with a standard solution of potassium ferro cyanide in the usual manner.

IRON & ALUMINUM

The filtrate from the Zinc carbonate which has

been evaporating on the hot plate is very carefully neutralized with ammonia and any aluminum which may have escaped precipitation in either of the other two cases, precipitated in the usual manner as a hydrate. All three hydroxide precipitates, "A", "B", and the one just obtained, are now placed in a weighed platinum crucible. After ignition a few drops of sulphuric acid and about 10 drops of hydrofluoric acid are added to rid the precipitate of the silica which has accumulated during the various steps. Heat to expell the last traces of sulphuric acid, ignite, cool and weigh as combined aluminum and iron oxides. To obtain the iron contents, pulverize the lumps of oxides with the blunt end of a stirring rod and fuse with a mixture of sodium and potassium carbonates. Dissolve the metal in a dilute solution of sulphuric acid and reduce with zinc in the usual manner, titrating the iron with a standard permanganate solution.

MODIFIED METHODS

As stated before, an alloy showing so complex a nature as the one just described is very rarely met with in commercial work. It has, therefore, been

found expedient in this laboratory to divise shorter schemes for the analyses of simpler combinations. Among the more common alloys may be mentioned babbitt metal consisting of 3 to 7 metals, e.g., tin, antimony, and copper in various proportions, up to the more complex babbitts which may consist of tin, antimony, copper, zinc, lead, nickel, bismuth, and perhaps a very small amount of phosphorus.

Brass in its simplest form consists of copper and zinc in one of the standard proportions depending upon the use to which it is put. Cast brass may have the following ingredients: Copper, zinc, iron, lead, and tin. The last three mentioned being, of course, very low in proportion to the other metals.

Bronze is essentially a mixture of copper and tin with small amounts of impurities such as iron, lead, etc., the composition varying as in the case of brass.

Another important mixture to be considered is the copper, zinc, nickel alloy, which product constitutes the metal known on the market as German Silver. In this alloy, as in the others, small amounts

of certain other metals may be present; principally, lead and iron.

The composition of solder varies a great deal, from the simplest form consisting of tin and lead to the very complex mixture containing tin, lead, and small amounts of antimony, zinc, bismuth, cadmium, aluminum, etc.

Bismuth and cadmium play an important part in the fusible alloys used in automatic sprinkling devices. The composition of these metals may vary from a simple alloy of cadmium and bismuth to more complicated mixtures containing tin, lead, cadmium, bismuth, and very small amounts of the higher melting elements.

It is not within the scope of this article to deal with the analyses of all these different compositions. It will, however, not be amiss to describe shorter methods for the determination of tin, antimony, copper, lead, iron, and aluminum which may be employed where the simplicity of the structure of the alloys permits.

TIN

This method is available where the lead content is less than 20% and no more than 5% of either copper, or nickel is present in the alloys.

Place a .5 to 1 gram sample is a 500 cc Jena Erlenmeyer flask, add if necessary .15 gram of antimony, cover the sample with 10 cc of concentrated sulphuric acid and heat to effect solution. Remove from the hot plate and cool. Dilute the cold sulphuric acid solution with 10 cc of water, add 105 cc of hydrochloric acid and then 78 cc more of water. Proceed with the reduction and titration exactly as described in the previous tin method.

ANTIMONY

This method is available where copper and nickel are below 5%. Some difficulty has been experienced at times in getting accurate results in the presence of large percentages of lead. It has been found advisable, therefore, when this metal is present in large quantities to use the regular method in preference to this one.

Place a 1/2 or 1 gram sample, depending on the

amount of antimony present, in a 500 cc Jena Erlenmeyer flask and cover with 10 cc of concentrated sulphuric acid. Place the container upon the hot plate and dissolve. When this has been accomplished, cool, and add 50 cc of water and 10 cc of concentrated hydrochloric acid. Replace over the source of heat and permit the contents to boil for a few seconds, cool, and add exactly 110 cc water. Continue from this point as described in the previous antimony analysis.

COPPER

The only metals which interfere in this method are bismuth and cadmium. Hence in the absence of these the following scheme is available:

Place one gram of the sample in a #2 beaker and add 40 cc of concentrated hydrochloric acid. When the first violent action has subsided add chlorate in small amounts until the solution is completed. Boil to remove the chlorine fumes and remove from the hot plate. Add 100 cc hot water and 8 grams of oxalic acid. Bring to a boil and if a white precipitate ensues add concentrated hydrochloric acid until the solution is clear. Add hot water, keeping

the solution sufficiently concentrated to prevent the precipitation of the tin and pass hydrogen sulphide until the separation of the sulphides is complete. Filter with suction, and wash well with hot water. Place the paper and precipitate in the original beaker, cover with 30 cc of water containing about 5 grams of potassium hydroxide and boil for a few minutes. Add 200 cc of hot water, filter with suction and wash the precipitate thoroughly to remove as much as possible the small amounts of tin and antimony which have been carried down mechanically with the precipitate. Place the two filter papers and copper sulphide in a porcelain crucible and ignite. Transfer the oxide to a #1 beaker, carefully removing all traces of copper from the crucible with hot concentrated nitric acid. Add a few cc of nitric acid to the oxides and concentrate to a volume of about 1 cc. From here the method is exactly similar to that described under copper in the original scheme.

LEAD

None of the elements treated in this paper have been found to interfere with this determination. In

case a rapid analysis is desired the following scheme is available regardless of the composition of the alloy, provided, of course, no other metals than those discussed in this article are present.

Effect the solution of the alloy with hydrochloric acid and potassium chlorate in the usual manner. Boil to expell the chlorine and dilute with 150 cc of cold water. In the absence of tin, zinc, bismuth, cadmium, etc., add phenolthalein; otherwise this is not necessary. Cool thoroughly and add a cold concentrated solution of potassium hydroxide until the solution turns pink or a precipitate of the metal hydroxides has been formed. Copper exhibits a great tendency to separate out in a hot strongly alkaline mixture, thereby coloring the solution black. Again cool and slowly add a concentrated solution containing 10 grams of potassium hydrate. It is obvious that a clear solution will not be obtained if cadmium and bismuth or iron are present and even in their absence a slight turbidity remains. The solution is diluted up with cold water and hydrogen sulphide introduced until all the metals with the excep-

tion of tin and antimony have been precipitated. Filter, avoiding the use of suction and wash with a weak solution of sodium sulphide. Even though the filtration is exceedingly slow, it is necessary to wash thoroughly, especially if the percentage of tin is very high; otherwise trouble may be experienced later due to the presence of an excessive amount of this element. Place the paper with the sulphides in a #2 beaker, add the usual amount of nitric and sulphuric acids and take to fumes. Cool, add 150 cc 1-1 alcohol and allow to stand for the required time after which the lead sulphate is filtered on a gooch crucible, ignited, and weighed. This method is very satisfactory provided care is taken in washing the sulphides thoroughly.

IRON & ALUMINUM

It is sometimes desirable to make a rapid determination of the iron and aluminum in which case the following method is available:

Make the usual sulphide separation and boil to expell the hydrogen sulphide. In the absence of lead add ammonium chloride and separate the iron and alum-

inum by the usual acetate method. In case this element is present it is necessary to evaporate to fumes of sulphur trioxide and filter off the lead sulphate before proceeding with this separation. Dissolve the precipitate in hot dilute hydrochloric acid and reprecipitate as hydrates. In the absence of nickel the acetate separation may be omitted and a double precipitation of the hydroxides substituted. It is essential in every case to add the necessary amount of ammonium chloride. The hydrates are placed in a platinum crucible, treated with sulphuric and hydrofluoric acids in the usual manner and weighed as oxides. Separate the iron and aluminum as described in the regular method.

F I N I S